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## PATENT ABSTRACTS OF JAPAN

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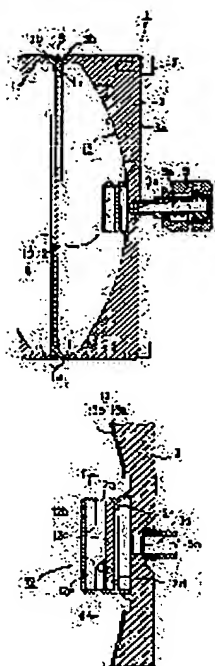
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(54) WATER CONTENT PRODUCING REACTOR



(57)Abstract:

PROBLEM TO BE SOLVED: To raise the water content producing reactivity to stably produce the water content under specified condition by providing an inlet reflector, filter and outlet reflector-diffuser in a reactor and providing a diffusion filter having a Pt coating in the outlet reflector-diffuser.

SOLUTION: A gas diffusing member 8 is composed of an inlet reflector, filter and outlet reflector-diffuser 12 which has a diffusion filter 12c and Pt coating 12d on the outer surface of the filter 12 at the space side. A Pt coating 13 provided on the inner wall of a reactor 1 top covert a non-radical H or O gas into radicals,

thus H and O are brought into reaction instantaneously to produce water. The coating 13 stably produces water continuously at 1000 cc/min or more at about 500°C over the entire area due to the reaction heat.

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## CLAIMS

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[Claim(s)]

[Claim 1] It is formed combining two furnace body members (2) and (3), and is punctured by the reactor body (1) and; one furnace body member (2) which have the space section (1a) inside. It is punctured by the furnace body member (3) of a gas supply path (2c) and; another side which introduces material gas to said space section (1a). The moisture gas outlet path which derives generation water from said space section (1a) (3c); It fixes to the space section side of a furnace body member (2) said the shape of a gas supply path (2c) and the same axle. On a wall surface, a bore (9c) The inside end face of a tubed case object (9a) and a case object (9a) which it has It fixes to the space section side of a furnace body member (3) said the shape of a moisture gas outlet path (3c) and the same axle. the entrance-side reflector (9) which consists of the reflecting plate (9b) to close, and; -- the filter (10) arranged in the space section (1a) of said reactor body (1), and; -- On a wall surface, a bore (12e) The tubed case object which it has The inside end face of (12a) and a case object (12a) The reflecting plate to close (12b) and a case object The diffusion filter arranged in the interior of (12a) The reactor for moisture generating constituted from a platinum coating coat (13) prepared in the internal surface of the outlet side echo and diffuser (12) which consists of (12c) and the platinum coating coat (12d) prepared in the diffusion filter (12c), and; reactor body (1), and;.

[Claim 2] The reactor for moisture generating according to claim 1 which used the filter (10) as the filter (10) which has a bore 200 micrometers or less.

[Claim 3] The reactor for moisture generating according to claim 1 which used the diffusion filter (12c) as the diffusion filter (12c) which has a bore 50 micrometers or more.

[Claim 4] The reactor for moisture generating according to claim 1 considered as the configuration which arranges a filter (10) in the center section of both the bodies member (2) and (3) while forming combining the furnace body member (2) which has the hollow part (2a) of the shape of a curvature side of isomorphism voice for a reactor body (1) mostly, and the furnace body member (3) which has a curvature side-like hollow part (3a) the letter of opposite.

## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to amelioration of the reactor for moisture generating used mainly by the semi-conductor manufacturing facility.

[0002]

[Description of the Prior Art] For example, with [ of the silicon by the moisture oxidation style in semi-conductor manufacture ] an oxide film, the super-high purity water exceeding 1000 cc/min at least is needed. therefore, this applicant develops the reactor for moisture generating of the structure which point-comes, boils and is shown in drawing 4 , and opens to the public as Japanese Patent Application No. No. 242246 [ eight to ].

[0003] The heat-resistant furnace body members 22 and 23 which the reactor body 21 of above-mentioned drawing 4 equipped with the joint 24 for gas supply, and the joint 25 for moisture gas fetch, Entrance-side reflecting plate 29a and outlet side reflecting plate 29b which were prepared in the interior of a reactor 21 gas supply path 24a of both the furnace body members 22 and 23 and moisture gas outlet path 25a, and in the shape of opposite, It is formed from the filter 30 prepared in the center of the interior of a reactor 21, and the platinum coating coat 32 grade prepared in the internal surface of the furnace body member 23. Moreover, said platinum coating coat 32 is formed by fixing platinum coat 32b with a vacuum evaporation method of construction, an ion plating method of construction, etc. on barrier layer anodic oxide coating 32a which consists of nitrides, such as TiN formed in the internal surface of the furnace body member 23.

[0004] <sup>\*\*</sup>(ing), the hydrogen and oxygen which were supplied to the interior of the reactor body 21 through gas supply path 24a are diffused by the member for diffusion which consists of entrance-side reflecting plate 29a, a filter 30, and outlet side reflecting plate 29b, and contact the platinum coating coat 32. Reactivity is raised by the catalysis of platinum and the oxygen and hydrogen in contact with the platinum coating coat 32 will be in the so-called condition of having been radical-ized, by it. The hydrogen and oxygen which were radical-ized react under temperature lower than the ignition temperature of hydrogen mixed gas in an instant, and they generate moisture, without carrying out elevated-temperature combustion.

[0005] The reactor body 21 of said drawing 4 can attain the large miniaturization of a moisture generator, can obtain the mixed gas of the high purity water of an amount and high purity water which exceed 1000 cc/min under moreover more high reactivity and responsibility, and oxygen, and attracts epoch-making attention in the field of a semi-conductor manufacturing technology.

[0006] what shows aging of moisture generating conversion [ in / in drawing 5 / the reactor body (70mm in outer diameter about 134mmphi and thickness, about 490 cc of content volume, moisture yield 1000 cc/min, the degree of coke oven temperature of about 400 degrees C) 21 of said drawing 4 ] -- it is -- material gas -- oxygen -- rich or hydrogen -- even if it is rich gas, under about 98.5 - 99.0% of moisture generating conversion, it is stabilized and water can be generated.

[0007] However, the temperature of the reactor body 21 will mix into the moisture which it is difficult moisture for about 400 degrees C or less and a moisture yield to raise said moisture generating conversion to about 99.0% or more under the condition of 1000 or more cc/min, and about 1% of unreacted oxygen and hydrogen generated. Consequently, only the pure water which contains neither hydrogen nor oxygen has the problem referred to as being unable to take out the pure water which does not contain hydrogen, and mixture of only oxygen.

[0008]

[Problem(s) to be Solved by the Invention] This invention makes it a technical problem to raise further the conversion of the hydrogen in the reactor body 21 of said drawing 4 , and oxygen, the temperature of the reactor body 21 is set at about 400 degrees C or less, and sets a moisture yield under the condition of 1000 or more cc/min, and uses as an offer plug the reactor for moisture generating which made it possible to acquire 99% or more of moisture generating conversion at stability and a long period of time.

[0009]

[Means for Solving the Problem] by the way, in the reactor body 21 of drawing 4 , as a cause which unreacted hydrogen and oxygen mix to moisture gas outlet path 25a \*\* When the platinum coating coat 32 is not contacted but oxygen and hydrogen reach to moisture gas outlet path 25a directly, \*\* Although 1 \*\* was radical-ized and two kinds in the case of returning to the condition before reaching to moisture gas outlet path 25a and being radical-ized here can be considered, without reacting with hydrogen or oxygen, the former case is overwhelmingly assumed [ many ].

[0010] According to an invention in this application person's etc. experimental result, when outlet side reflecting plate 29b is removed by the reactor body 21 of drawing 4 , as shown in drawing 6 , the moisture [ temperature / of a reactor / yield / 400 degrees C and / moisture / whenever / 500 cc/min and overgas ] generating conversion under the conditions of 0 becomes about 91%. Although this conversion is not data under the same conditions at all since moisture yields differ, it serves as a value low about about 7% as compared with the moisture generating conversion (about 98%) in the case of said drawing 5 .

[0011] When there is no outlet side reflecting plate 29b, this shows that it reaches to moisture gas outlet path 25a directly, without radical-izing oxygen and hydrogen of a considerable amount, and shows that improvement in moisture generating conversion is possible by adding amelioration to outlet side reflecting plate 29b.

[0012] Moreover, when there is no outlet side reflecting plate 29b so that clearly also from said drawing 6 , moisture generating conversion falls, so that material gas becomes hydrogen Rich. For example, in the moisture yield of 400 degrees C and 500 cc/min, to moisture generating conversion being about 86%, when hydrogen is rich 100%, reactor temperature becomes about 97%, when oxygen is rich 100%, and about about 11% of difference arises among both.

[0013] That is, it is assumed that the oxygen which reaches to moisture gas outlet path 25a, without oxygen's accompanying to straight-line [ of hydrogen ]-like flow in being comparatively easy to diffuse the direction of oxygen in the interior of the reactor body 21 of structure like drawing 4 , being comparatively hard to diffuse the direction of hydrogen to a thing with small linear performance traverse, and being hydrogen Rich's material gas, since linear performance traverse is high, and being radical-ized increases.

[0014] then, the thing for which this artificer raises the diffusibility of the gas of outlet side reflecting plate 29b, especially the diffusibility over hydrogen in the reactor body 21 of drawing 4 -- oxygen -- not only rich material gas but hydrogen -- also in the case of rich material gas, it hit on an idea of the ability of moisture generating conversion to be made higher than about 98 - 99% of conversion in the case of drawing 5 . Moreover, while manufacturing the outlet side reflecting plate and the diffusion plate of various kinds of structures based on this idea, much moisture generating trials were performed using this.

[0015] The invention in this application is created from the result of the idea like \*\*\*\*, and the moisture generating trial based on this. Invention according to claim 1 It is formed combining the furnace body member 2 and the furnace body member 3, and is punctured by the reactor body 1 and; furnace body member 2 which have space section 1a inside. It is punctured by gas supply path 2c and; furnace body member 3 which introduce material gas to said space section 1a. Moisture gas outlet path 3c which derives generation water from said space section 1a; It fixes to the space section side of the furnace body member 2 said the shape of moisture gas outlet path 2c and the same axle. The entrance-side reflecting plate 9 which changes from reflecting plate 9b which closes the inside end face of tubed case object 9a and case object 9a which has bore 9c to a wall surface, and the filter 10 arranged in space section 1a of the; aforementioned reactor body 1; It fixes to the space section side of the furnace body member 3 said the

shape of moisture gas outlet path 3c and the same axle. On a wall surface, bore 12e The inside end face of tubed case object 12a and case object 12a which it has The platinum coating coat 13 prepared in the internal surface of the outlet side echo and the diffuser 12 which consists of 12d of platinum coating coats prepared in reflecting plate 12b to close, diffusion filter 12c arranged in the interior of case object 12a, and diffusion filter 12c, and; reactor body 1 It considers as the requirements for a configuration of invention. [0016] Invention according to claim 2 uses the filter 10 in invention of claim 1 as the filter 10 which has a bore 200 micrometers or less.

[0017] Invention according to claim 3 sets diffusion filter 12c in invention of claim 1 to diffusion filter 12c which has a bore 50 micrometers or more.

[0018] Invention according to claim 4 is taken as the configuration which arranges a filter 10 in the center section of both the bodies members 2 and 3 while forming it, combining the furnace body member 2 which has hollow part 2a of the shape of a curvature side of isomorphism voice for the reactor body 1 in invention of claim 1 mostly, and the furnace body member 3 which has curvature side-like hollow part 3a the letter of opposite.

[0019]

[Embodiment of the Invention] Hereafter, the embodiment of this invention is explained based on a drawing. Drawing 1 is drawing of longitudinal section of the reactor body for moisture generating concerning this invention. Moreover, drawing 2 is the expanded sectional view of an outlet side echo and diffuser, and drawing 3 is I-I \*\*\*\*\* of drawing 2. In drawing 1 1 a furnace body member and 4 for a reactor body, and 2 and 3 The joint for gas supply, 5 a filter flange and 7 for the joint for moisture gas fetch, and 6 The bolt for reactor anchoring, In 8, the member for gaseous diffusion and 9 a filter and 11 for an entrance-side reflector and 10 The filter carrier piece of a filter flange, 12 is an outlet side echo and diffuser, 13 is a platinum coating coat, and the reactor 1 is formed in the short cylindrical shape by welding two furnace body members 2 and 3 made from stainless steel mostly formed in isomorphism voice in the shape of an airtight.

[0020] The base is established for curvature side-like hollow part 2a in the interior, and, as for one [ said ] furnace body member 2, gas supply path 2c is further drilled in the center section. Moreover, the joint 4 for gas supply is formed in the lateral surface, and gas supply path 4a of the joint 4 for gas supply formed in this lateral surface is opened for free passage into hollow part 2a. Similarly, the base is established for curvature side-like hollow part 3a in the interior, and, as for the furnace body member 3 of another side, gas supply path 3c is further drilled in the center section. Moreover, the joint 5 for moisture gas fetch is formed in the lateral surface, and moisture gas outlet path 5a of

the joint 5 for moisture gas fetch formed in this lateral surface is opened for free passage into hollow part 3a.

[0021] Flange object 2b and 3b are formed in the medial surface of said both furnace body members 2 and 3, respectively, and the reactor body 1 which has space section 1a inside is constituted by carrying out welding immobilization of both flange object 2b and the 3b through the filter flange 6 at the shape of an airtight. In addition, in drawing 1, it is good also as a configuration which is considering as the configuration which fixes both flange object 2b and 3b by welding, or interposes a gasket (graphic display abbreviation) and enables assembly fixing of the dissociation of both flange object 2b and 3b by a clamp (graphic display abbreviation) etc. Moreover, although both the bodies members 2 and 3 are mostly formed in the thing of the same configuration in drawing 1, of course, one side may be formed in the gestalt of the tube-like object of an owner bottom at the gestalt of the shape of a flange which closes opening of a tube-like object for another side.

[0022] Said member 8 for gaseous diffusion is formed from the entrance-side reflecting plate 9, the filter 10, and the outlet side echo / diffusion plate 12 grade, and as shown in drawing 1, it is arranged in the interior of the reactor body 1. That is, the entrance-side reflecting plate 9 is formed from short tubed case object 9a and reflecting plate 9b which closes the inside end face of case object 9a, and bore 9c is formed in the peripheral wall of case object 9a. In addition, the entrance-side reflecting plate 9 concerned is arranged this and in the shape of the same axle in gas supply path 2c of the base of the furnace body member 2, and the location which counters, and welding fixing is carried out at this.

[0023] Moreover, said filter 10 is formed from the filter made from stainless steel which has bore about 200 micrometers or less, and the filter which has the bore of the shape of an average of 2-micrometer mesh is used with this operation gestalt. In addition, the filter flange 6 made from stainless steel is welded to the periphery edge of a filter 10, and welding immobilization of the filter 10 is carried out through this filter flange 6 to the furnace body members 2 and 3.

[0024] As shown in drawing 2 R> 2 and drawing 3, said outlet side echo and diffuser 12 Furthermore, short cylinder-like case object 12a, Reflecting plate 12b which closes the inside end face of case object 12a, and diffusion filter 12c, It is formed from 12d of platinum coating coats prepared in the outside surface by the side of the space section of diffusion filter 12c, 12f of filter presser feet etc., etc., and two or more bore 12e is drilled by the peripheral wall of the inside part of case body 12a. That is, all of said case object 12a, reflecting plate 12b, etc. are formed with stainless steel, and reflecting plate 12b



has fixed by spot welding to case object 12a. Moreover, diffusion filter 12c is formed from the filter made from stainless steel which has a bore 50 micrometers or more.

[0025] 12d of platinum coating coats with a thickness of 0.2-8 micrometers is formed in the outside surface by the side of the space section of said diffusion filter 12c. Namely, the 12d of the platinum coating coats concerned is formed from the barrier layer anodic oxide coating with a thickness of 0.1-5 micrometers formed in the outside surface of diffusion filter 12c made from TiN, and a platinum coat with a thickness of 0.1-3 micrometers formed on it. In order to prevent that blinding arises in diffusion filter 12c by formation of 12d of platinum coating coats, The bore of the shape of a mesh of the filter made from stainless steel which constitutes diffusion filter 12c is selected by the magnitude (at this operation gestalt, they are 70 micrometers and 200 micrometers) of 50 micrometers or more.

[0026] In addition, about the formation approach of 12d of platinum coating coats, since it is the same as that of the case of the platinum coating coat 13 prepared in the internal surface of the reactor body 1 mentioned later, detailed explanation is omitted here. Moreover, although he is trying to form 12d of platinum coating coats in the outside surface by the side of the space section of diffusion filter 12c with this operation gestalt, it is also possible to prepare 12d of platinum coating coats in the interior of diffusion filter 12c. Furthermore, said outlet side echo and diffuser 12 are arranged in the shape of the same axle in moisture gas outlet path 5a of the base of the furnace body member 3, and the location which counters, and welding fixing is carried out at this.

[0027] Although each base of the furnace body members 2 and 3 is made into the shape of a curvature side, you may make it form this in a plane base in said drawing 1 . Moreover, in drawing 1 , it is also possible to enlarge the linear dimension concerned and to press down the capacity of the depth dimension of about 1/6 and hollow part 3a of the depth dimension of hollow part 2a which penetrates the core of a filter 10 for the linear dimension of the entrance-side reflector 9, or the outlet side echo and diffuser 12, although about 1/is set to 3. Furthermore, although the filter which is a disk mold as a filter 10, and made the whole surface the gas transparency section is used, it changes to this, and it is a disk mold and you may make it use the filter of a configuration of having made only the peripheral face section into the filter section (gas transparency section) in drawing 1 .

[0028] After forming said platinum coating coat 13 throughout the internal surface of the furnace body member 3 made from SUS316L and forming barrier layer anodic oxide coating 13a made from TiN in the internal surface of the furnace body member 3 first, platinum coat 13b is formed on it. Moreover, the thickness of barrier layer anodic oxide

coating 13a has 0.1 micrometers - optimal about 5 micrometers, and barrier layer anodic oxide coating 13a made from TiN with a thickness of about 2 micrometers is formed by the ion plating method of construction in drawing 1 . Furthermore, 0.1 micrometers - about 3 micrometer of \*\* are suitable for the thickness of said platinum coat 13b, and platinum coat 13b with a thickness of about 1 micrometer is formed by the vacuum deposition method in drawing 1 .

[0029] In addition, as the formation approach of barrier layer anodic oxide coating 13a, it is also possible to use PVD, such as the ion sputtering method and a vacuum deposition method, chemical vapor deposition (CVD method), hot pressing, a spraying process, etc. in addition to said ion plating method of construction. Moreover, the formation approach of platinum coat 13b has an ion plating method of construction, the ion sputtering method, chemical vapor deposition, usable hot pressing, etc. in addition to said vacuum evaporation technique, and is still more nearly usable at the time of the matter with which conductivity, such as TiN, has barrier layer anodic oxide coating 13a. [ of plating ]

[0030] the gas injected into case object 9a of the entrance-side reflector 9 through gas supply path 4a of the joint 4 for gas supply being injected through bore 9c prepared in the peripheral wall after colliding to reflecting plate 9b, and being spread within hollow part 2a -- a filter 10 -- it passes through the whole surface uniformly mostly, and enters into hollow part 3a of the furnace body member 3. moreover, the mixed gas of the hydrogen injected into said hollow part 3a, and oxygen -- the whole surface of the platinum coating coat 13 -- continuing -- equal -- collision contact -- carrying out -- thereby -- being the so-called -- it will be \*\*\*\*\*-ized. Furthermore, the hydrogen and oxygen which were activated react mainly within hollow part 3a in an instant, and generate water. And the moisture gas formed mainly by hollow part 3a is drawn to moisture gas outlet path 5a through bore 12e of an outlet side echo and diffuser 12, and diffusion filter 12c, and goes.

[0031] by the way, the thing which the great portion of hydrogen which penetrated the filter 10 and entered into hollow part 3a, and oxygen gas collide and contact with platinum coat 13b -- a radical -- the hydrogen and oxygen which were-izing [ oxygen ] and radical-ized -- the -- the whole quantity is mostly changed into water in response to an instant. Moreover, although a part of hydrogen which entered into hollow part 3a, and oxygen gas may go straight on as it is, the hydrogen and oxygen gas which went [ this ] straight on are collided and re-diffused to reflecting plate 12b. Consequently, the hydrogen and oxygen which reach to diffusion filter 12c through bore 12e in the platinum coat 13b and non-contact state decrease substantially.

[0032] On the other hand, in this invention, diffusion filter 12c which prepared 12d of platinum coating coats in an outlet side echo and diffuser 12 is prepared. Therefore, said platinum coat 13b, the hydrogen which reached to the way among case body 12a through bore 12e in the non-contact state, and oxygen gas become that there is almost no bypassing into moisture gas outlet path 3c as it is, and it is radical-ized by contacting 12d of platinum coating coats. That is, while hydrogen and oxygen gas under the condition of radical[ non]-izing are radical-ized by 12d of platinum coating coats of said diffusion filter 12c and hydrogen and oxygen of a non-radical-ized condition will be in the condition of \*\*\*\*\* 0, the hydrogen and oxygen which were radical-ized react in an instant, and water is generated.

[0033] moreover, the hydrogen and oxygen in the condition of the probability bypassed into moisture gas outlet path 3c while the hydrogen and oxygen in the condition of having been radical-ized when diffusion filter 12c existed in a way among said case bodies 12 have been unreacted having become smaller, and having been radical-ized -- all will contribute to a moisture generation reaction mostly.

[0034] In addition, that the platinum coating coat 13 is locally heated by heat of reaction by forming the member 8 for gaseous diffusion which consists of said entrance-side reflector 9, a filter 10, and an outlet side echo and diffuser 12 in a reactor body becomes that there is nothing. It is proved that moisture generating can be performed where the whole region is mostly held to the temperature of the platinum coating coat 13 in which about about 500 degrees is, and it moreover continues under the high moisture generating conversion and the responsibility exceeding about 99% at insurance, and water generating of the amount of 1000 or more cc/min can be performed.

[0035]

[Example] In the reactor body 1 of drawing 1 , the dimension of the furnace body members 2 and 3 was made into diameter 134mmphi and the product with a thickness of 33.5mm made from SUS316L, and the curvature side of hollow parts 2a and 3a was made into the curvature side with a radius of curvature of R= 108mm. Moreover, the filter (about 1.7mm in thickness) which has an average of 2.0-micrometer bore which carried out two or more sheet laminating of the mesh made from stainless steel as a filter 10 was used. That whose height the outer diameter of case object 9a is 22mmphi, and is 5mm as an entrance-side reflector 9 again furthermore, as an outlet side echo and diffuser 12 22mmphi and height 10.5mm, [ the outer diameter of case object 12a ] Diffusion filter 12c used the thing of a filter (about 1.7mm in thickness) which has an average of 200-micrometer bore which carried out two or more sheet laminating of the mesh made from a filter (about 1.7mm in thickness), and stainless steel which has an

average of 70-micrometer bore which carried out two or more sheet laminating of the mesh made from stainless steel. In addition, 12d of platinum coating coats of diffusion filter 12c forms a platinum coat with a thickness of about 2 micrometers on the barrier layer anodic oxide coating made from TiN with a thickness of about 2 micrometers.

[0036] What formed barrier layer anodic oxide coating (about 2-micrometer [ in thickness ], ion plating method) 13a made from TiN in the internal surface of the furnace body member 3, and, on the other hand, formed with a thickness of about 1 micrometer platinum coat (vacuum deposition method) 13b on it as a platinum coating coat 13 was used.

[0037] Moisture generating conversion was searched for by supplying the material gas of  $H_2$  1000 cc/min+O $_2$  1000 cc/min from gas supply path 4a,  $H_2$  1000 cc/min+O $_2$  500 cc/min, and  $H_2$  1500 cc/min+O $_2$  500 cc/min, and surveying the moisture which flows out of moisture gas outlet path 5a using the above-mentioned reactor for moisture generating. Consequently, even if it was in which case of the aforementioned  $H_2$ ,  $H_2$ , and  $H_2$ , in the continuation moisture generating trial over about 10 hours, 99.3% or more of moisture generating conversion was acquired.

[0038]

[Effect of the Invention] This invention is considered as the configuration which prepares the diffusion filter equipped with the platinum coating coat in the interior of an outlet side echo and diffuser while it prepares an entrance-side reflector, a filter, and an outlet side echo and diffuser in the interior of a reactor body as above-mentioned. Consequently, the unconverted gas which flows out into a moisture gas outlet path almost serves as zero, and, also in the case of material gas of hydrogen Rich, 99.0% or more of high moisture generating conversion is acquired not to mention the case of oxygen Rich's material gas. Moreover, with heat of reaction, it becomes that there is also no being heated locally, and under the temperature of about about 500 degrees C, the platinum coating coat within a reactor body can be stabilized, and can generate the moisture of 1000 or more cc/min.

[0039] In invention of claim 4, it is considering as the configuration which forms a reactor body mostly, combining the furnace body member of the same configuration the letter of opposite. Consequently, the structure of a reactor body is simplified and reduction with a sharp manufacturing cost becomes possible. This invention does so the practical use which was excellent as above-mentioned.

#### TECHNICAL FIELD

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[Industrial Application] This invention relates to amelioration of the reactor for

moisture generating used mainly by the semi-conductor manufacturing facility.

#### PRIOR ART

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[Description of the Prior Art] For example, with [ of the silicon by the moisture oxidation style in semi-conductor manufacture ] an oxide film, the super-high purity water exceeding 1000 cc/min at least is needed. therefore, this applicant develops the reactor for moisture generating of the structure which point-comes, boils and is shown in drawing 4 , and opens to the public as Japanese Patent Application No. No. 242246 [ eight to ].

[0003] The heat-resistant furnace body members 22 and 23 which the reactor body 21 of above-mentioned drawing 4 equipped with the joint 24 for gas supply, and the joint 25 for moisture gas fetch, Entrance-side reflecting plate 29a and outlet side reflecting plate 29b which were prepared in the interior of a reactor 21 gas supply path 24a of both the furnace body members 22 and 23 and moisture gas outlet path 25a, and in the shape of opposite, It is formed from the filter 30 prepared in the center of the interior of a reactor 21, and the platinum coating coat 32 grade prepared in the internal surface of the furnace body member 23. Moreover, said platinum coating coat 32 is formed by fixing platinum coat 32b with a vacuum evaporation method of construction, an ion plating method of construction, etc. on barrier layer anodic oxide coating 32a which consists of nitrides, such as TiN formed in the internal surface of the furnace body member 23.

[0004] \*(ing), the hydrogen and oxygen which were supplied to the interior of the reactor body 21 through gas supply path 24a are diffused by the member for diffusion which consists of entrance-side reflecting plate 29a, a filter 30, and outlet side reflecting plate 29b, and contact the platinum coating coat 32. Reactivity is raised by the catalysis of platinum and the oxygen and hydrogen in contact with the platinum coating coat 32 will be in the so-called condition of having been radical-ized, by it. The hydrogen and oxygen which were radical-ized react under temperature lower than the ignition temperature of hydrogen mixed gas in an instant, and they generate moisture, without carrying out elevated-temperature combustion.

[0005] The reactor body 21 of said drawing 4 can attain the large miniaturization of a moisture generator, can obtain the mixed gas of the high purity water of an amount and high purity water which exceed 1000 cc/min under moreover more high reactivity and responsibility, and oxygen, and attracts epoch-making attention in the field of a semi-conductor manufacturing technology.

[0006] what shows aging of moisture generating conversion [ in / in drawing 5 / the reactor body (70mm in outer-diameter about 134mmphi and thickness, about 490 cc of

content volume, moisture yield 1000 cc/min, the degree of coke oven temperature of about 400 degrees C) 21 of said drawing 4 ] -- it is -- material gas -- oxygen -- rich or hydrogen -- even if it is rich gas, under about 98.5 - 99.0% of moisture generating conversion, it is stabilized and water can be generated.

[0007] However, the temperature of the reactor body 21 will mix into the moisture which it is difficult moisture for about 400 degrees C or less and a moisture yield to raise said moisture generating conversion to about 99.0% or more under the condition of 1000 or more cc/min, and about 1% of unreacted oxygen and hydrogen generated. Consequently, only the pure water which contains neither hydrogen nor oxygen has the problem referred to as being unable to take out the pure water which does not contain hydrogen, and mixture of only oxygen.

#### EFFECT OF THE INVENTION

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[Effect of the Invention] This invention is considered as the configuration which prepares the diffusion filter equipped with the platinum coating coat in the interior of an outlet side echo and diffuser while it prepares an entrance-side reflector, a filter, and an outlet side echo and diffuser in the interior of a reactor body as above-mentioned. Consequently, the unconverted gas which flows out into a moisture gas outlet path almost serves as zero, and, also in the case of material gas of hydrogen Rich, 99.0% or more of high moisture generating conversion is acquired not to mention the case of oxygen Rich's material gas. Moreover, with heat of reaction, it becomes that there is also no being heated locally, and under the temperature of about about 500 degrees C, the platinum coating coat within a reactor body can be stabilized, and can generate the moisture of 1000 or more cc/min.

[0039] In invention of claim 4, it is considering as the configuration which forms a reactor body mostly, combining the furnace body member of the same configuration the letter of opposite. Consequently, the structure of a reactor body is simplified and reduction with a sharp manufacturing cost becomes possible. This invention does so the practical use which was excellent as above-mentioned.

#### TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] This invention makes it a technical problem to raise further the conversion of the hydrogen in the reactor body 21 of said drawing 4 , and oxygen, the temperature of the reactor body 21 is set at about 400 degrees C or less, and sets a moisture yield under the condition of 1000 or more cc/min, and uses as an offer plug the reactor for moisture generating which made it possible to acquire 99% or

more of moisture generating conversion at stability and a long period of time.

#### MEANS

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[Means for Solving the Problem] by the way, in the reactor body 21 of drawing 4 , as a cause which unreacted hydrogen and oxygen mix to moisture gas outlet path 25a \*\* When the platinum coating coat 32 is not contacted but oxygen and hydrogen reach to moisture gas outlet path 25a directly, \*\* Although 1 \*\* was radical-ized and two kinds in the case of returning to the condition before reaching to moisture gas outlet path 25a and being radical-ized here can be considered, without reacting with hydrogen or oxygen, the former case is overwhelmingly assumed [ many ].

[0010] According to an invention in this application person's etc. experimental result, when outlet side reflecting plate 29b is removed by the reactor body 21 of drawing 4 , as shown in drawing 6 , the moisture [ temperature / of a reactor / yield / 400 degrees C and / moisture / whenever / 500 cc/min and overgas ] generating conversion under the conditions of 0 becomes about 91%. Although this conversion is not data under the same conditions at all since moisture yields differ, it serves as a value low about about 7% as compared with the moisture generating conversion (about 98%) in the case of said drawing 5 .

[0011] When there is no outlet side reflecting plate 29b, this shows that it reaches to moisture gas outlet path 25a directly, without radical-izing oxygen and hydrogen of a considerable amount, and shows that improvement in moisture generating conversion is possible by adding amelioration to outlet side reflecting plate 29b.

[0012] Moreover, when there is no outlet side reflecting plate 29b so that clearly also from said drawing 6 , moisture generating conversion falls, so that material gas becomes hydrogen Rich. For example, in the moisture yield of 400 degrees C and 500 cc/min, to moisture generating conversion being about 86%, when hydrogen is rich 100%, reactor temperature becomes about 97%, when oxygen is rich 100%, and about about 11% of difference arises among both.

[0013] That is, it is assumed that the oxygen which reaches to moisture gas outlet path 25a, without oxygen's accompanying to straight-line [ of hydrogen ]-like flow in being comparatively easy to diffuse the direction of oxygen in the interior of the reactor body 21 of structure like drawing 4 , being comparatively hard to diffuse the direction of hydrogen to a thing with small linear performance traverse, and being hydrogen Rich's material gas, since linear performance traverse is high, and being radical-ized increases.

[0014] then, the thing for which this artificer raises the diffusibility of the gas of outlet side reflecting plate 29b, especially the diffusibility over hydrogen in the reactor body 21

of drawing 4 -- oxygen -- not only rich material gas but hydrogen -- also in the case of rich material gas, it hit on an idea of the ability of moisture generating conversion to be made higher than about 98 - 99% of conversion in the case of drawing 5 . Moreover, while manufacturing the outlet side reflecting plate and the diffusion plate of various kinds of structures based on this idea, much moisture generating trials were performed using this.

[0015] The invention in this application is created from the result of the idea like \*\*\*\*, and the moisture generating trial based on this. Invention according to claim 1 It is formed combining the furnace body member 2 and the furnace body member 3, and is punctured by the reactor body 1 and; furnace body member 2 which have space section 1a inside. It is punctured by gas supply path 2c and; furnace body member 3 which introduce material gas to said space section 1a. Moisture gas outlet path 3c which derives generation water from said space section 1a; It fixes to the space section side of the furnace body member 2 said the shape of moisture gas outlet path 2c and the same axle. The entrance-side reflecting plate 9 which changes from reflecting plate 9b which closes the inside end face of tubed case object 9a and case object 9a which has bore 9c to a wall surface, and the filter 10 arranged in space section 1a of the; aforementioned reactor body 1; It fixes to the space section side of the furnace body member 3 said the shape of moisture gas outlet path 3c and the same axle. On a wall surface, bore 12e The inside end face of tubed case object 12a and case object 12a which it has The platinum coating coat 13 prepared in the internal surface of the outlet side echo and the diffuser 12 which consists of 12d of platinum coating coats prepared in reflecting plate 12b to close, diffusion filter 12c arranged in the interior of case object 12a, and diffusion filter 12c, and; reactor body 1 It considers as the requirements for a configuration of invention.

[0016] Invention according to claim 2 uses the filter 10 in invention of claim 1 as the filter 10 which has a bore 200 micrometers or less.

[0017] Invention according to claim 3 sets diffusion filter 12c in invention of claim 1 to diffusion filter 12c which has a bore 50 micrometers or more.

[0018] Invention according to claim 4 is taken as the configuration which arranges a filter 10 in the center section of both the bodies members 2 and 3 while forming it, combining the furnace body member 2 which has hollow part 2a of the shape of a curvature side of isomorphism voice for the reactor body 1 in invention of claim 1 mostly, and the furnace body member 3 which has curvature side-like hollow part 3a the letter of opposite.

[0019]

[Embodiment of the Invention] Hereafter, the embodiment of this invention is explained



based on a drawing. Drawing 1 is drawing of longitudinal section of the reactor body for moisture generating concerning this invention. Moreover, drawing 2 is the expanded sectional view of an outlet side echo and diffuser, and drawing 3 is I-I \*\*\*\*\* of drawing 2. In drawing 1 1 a furnace body member and 4 for a reactor body, and 2 and 3 The joint for gas supply, 5 a filter flange and 7 for the joint for moisture gas fetch, and 6 The bolt for reactor anchoring, In 8, the member for gaseous diffusion and 9 a filter and 11 for an entrance-side reflector and 10 The filter carrier piece of a filter flange, 12 is an outlet side echo and diffuser, 13 is a platinum coating coat, and the reactor 1 is formed in the short cylindrical shape by welding two furnace body members 2 and 3 made from stainless steel mostly formed in isomorphism voice in the shape of an airtight.

[0020] The base is established for curvature side-like hollow part 2a in the interior, and, as for one [ said ] furnace body member 2, gas supply path 2c is further drilled in the center section. Moreover, the joint 4 for gas supply is formed in the lateral surface, and gas supply path 4a of the joint 4 for gas supply formed in this lateral surface is opened for free passage into hollow part 2a. Similarly, the base is established for curvature side-like hollow part 3a in the interior, and, as for the furnace body member 3 of another side, gas supply path 3c is further drilled in the center section. Moreover, the joint 5 for moisture gas fetch is formed in the lateral surface, and moisture gas outlet path 5a of the joint 5 for moisture gas fetch formed in this lateral surface is opened for free passage into hollow part 3a.

[0021] Flange object 2b and 3b are formed in the medial surface of said both furnace body members 2 and 3, respectively, and the reactor body 1 which has space section 1a inside is constituted by carrying out welding immobilization of both flange object 2b and the 3b through the filter flange 6 at the shape of an airtight. In addition, in drawing 1, it is good also as a configuration which is considering as the configuration which fixes both flange object 2b and 3b by welding, or interposes a gasket (graphic display abbreviation) and enables assembly fixing of the dissociation of both flange object 2b and 3b by a clamp (graphic display abbreviation) etc. Moreover, although both the bodies members 2 and 3 are mostly formed in the thing of the same configuration in drawing 1, of course, one side may be formed in the gestalt of the tube-like object of an owner bottom at the gestalt of the shape of a flange which closes opening of a tube-like object for another side.

[0022] Said member 8 for gaseous diffusion is formed from the entrance-side reflecting plate 9, the filter 10, and the outlet side echo / diffusion plate 12 grade, and as shown in drawing 1, it is arranged in the interior of the reactor body 1. That is, the entrance-side reflecting plate 9 is formed from short tubed case object 9a and reflecting plate 9b which

closes the inside end face of case object 9a, and bore 9c is formed in the peripheral wall of case object 9a. In addition, the entrance-side reflecting plate 9 concerned is arranged this and in the shape of the same axle in gas supply path 2c of the base of the furnace body member 2, and the location which counters, and welding fixing is carried out at this.

[0023] Moreover, said filter 10 is formed from the filter made from stainless steel which has bore about 200 micrometers or less, and the filter which has the bore of the shape of an average of 2-micrometer mesh is used with this operation gestalt. In addition, the filter flange 6 made from stainless steel is welded to the periphery edge of a filter 10, and welding immobilization of the filter 10 is carried out through this filter flange 6 to the furnace body members 2 and 3.

[0024] As shown in drawing 2 R> 2 and drawing 3 , said outlet side echo and diffuser 12 Furthermore, short cylinder-like case object 12a, Reflecting plate 12b which closes the inside end face of case object 12a, and diffusion filter 12c, It is formed from 12d of platinum coating coats prepared in the outside surface by the side of the space section of diffusion filter 12c, 12f of filter presser feet etc., etc., and two or more bore 12e is drilled by the peripheral wall of the inside part of case body 12a. That is, all of said case object 12a, reflecting plate 12b, etc. are formed with stainless steel, and reflecting plate 12b has fixed by spot welding to case object 12a. Moreover, diffusion filter 12c is formed from the filter made from stainless steel which has a bore 50 micrometers or more.

[0025] 12d of platinum coating coats with a thickness of 0.2-8 micrometers is formed in the outside surface by the side of the space section of said diffusion filter 12c. Namely, the 12d of the platinum coating coats concerned is formed from the barrier layer anodic oxide coating with a thickness of 0.1-5 micrometers formed in the outside surface of diffusion filter 12c made from TiN, and a platinum coat with a thickness of 0.1-3 micrometers formed on it. In order to prevent that blinding arises in diffusion filter 12c by formation of 12d of platinum coating coats, The bore of the shape of a mesh of the filter made from stainless steel which constitutes diffusion filter 12c is selected by the magnitude (at this operation gestalt, they are 70 micrometers and 200 micrometers) of 50 micrometers or more.

[0026] In addition, about the formation approach of 12d of platinum coating coats, since it is the same as that of the case of the platinum coating coat 13 prepared in the internal surface of the reactor body 1 mentioned later, detailed explanation is omitted here. Moreover, although he is trying to form 12d of platinum coating coats in the outside surface by the side of the space section of diffusion filter 12c with this operation gestalt, it is also possible to prepare 12d of platinum coating coats in the interior of diffusion

filter 12c. Furthermore, said outlet side echo and diffuser 12 are arranged in the shape of the same axle in moisture gas outlet path 5a of the base of the furnace body member 3, and the location which counters, and welding fixing is carried out at this.

[0027] Although each base of the furnace body members 2 and 3 is made into the shape of a curvature side, you may make it form this in a plane base in said drawing 1 . Moreover, in drawing 1 , it is also possible to enlarge the linear dimension concerned and to press down the capacity of the depth dimension of about 1/6 and hollow part 3a of the depth dimension of hollow part 2a which penetrates the core of a filter 10 for the linear dimension of the entrance-side reflector 9, or the outlet side echo and diffuser 12, although about 1/is set to 3. Furthermore, although the filter which is a disk mold as a filter 10, and made the whole surface the gas transparency section is used, it changes to this, and it is a disk mold and you may make it use the filter of a configuration of having made only the peripheral face section into the filter section (gas transparency section) in drawing 1 .

[0028] After forming said platinum coating coat 13 throughout the internal surface of the furnace body member 3 made from SUS316L and forming barrier layer anodic oxide coating 13a made from TiN in the internal surface of the furnace body member 3 first, platinum coat 13b is formed on it. Moreover, the thickness of barrier layer anodic oxide coating 13a has 0.1 micrometers - optimal about 5 micrometers, and barrier layer anodic oxide coating 13a made from TiN with a thickness of about 2 micrometers is formed by the ion plating method of construction in drawing 1 . Furthermore, 0.1 micrometers - about 3 micrometer of \*\* are suitable for the thickness of said platinum coat 13b, and platinum coat 13b with a thickness of about 1 micrometer is formed by the vacuum deposition method in drawing 1 .

[0029] In addition, as the formation approach of barrier layer anodic oxide coating 13a, it is also possible to use PVD, such as the ion sputtering method and a vacuum deposition method, chemical vapor deposition (CVD method), hot pressing, a spraying process, etc. in addition to said ion plating method of construction. Moreover, the formation approach of platinum coat 13b has an ion plating method of construction, the ion sputtering method, chemical vapor deposition, usable hot pressing, etc. in addition to said vacuum evaporation technique, and is still more nearly usable at the time of the matter with which conductivity, such as TiN, has barrier layer anodic oxide coating 13a. [ of plating ]

[0030] the gas injected into case object 9a of the entrance-side reflector 9 through gas supply path 4a of the joint 4 for gas supply being injected through bore 9c prepared in the peripheral wall after colliding to reflecting plate 9b, and being spread within hollow

part 2a -- a filter 10 -- it passes through the whole surface uniformly mostly, and enters into hollow part 3a of the furnace body member 3. moreover, the mixed gas of the hydrogen injected into said hollow part 3a, and oxygen -- the whole surface of the platinum coating coat 13 -- continuing -- equal -- collision contact -- carrying out -- thereby -- being the so-called -- it will be \*\*\*\*\*-ized. Furthermore, the hydrogen and oxygen which were activated react mainly within hollow part 3a in an instant, and generate water. And the moisture gas formed mainly by hollow part 3a is drawn to moisture gas outlet path 5a through bore 12e of an outlet side echo and diffuser 12, and diffusion filter 12c, and goes.

[0031] by the way, the thing which the great portion of hydrogen which penetrated the filter 10 and entered into hollow part 3a, and oxygen gas collide and contact with platinum coat 13b -- a radical -- the hydrogen and oxygen which were-izing [ oxygen ] and radical-ized -- the -- the whole quantity is mostly changed into water in response to an instant. Moreover, although a part of hydrogen which entered into hollow part 3a, and oxygen gas may go straight on as it is, the hydrogen and oxygen gas which went [ this ] straight on are collided and re-diffused to reflecting plate 12b. Consequently, the hydrogen and oxygen which reach to diffusion filter 12c through bore 12e in the platinum coat 13b and non-contact state decrease substantially.

[0032] On the other hand, in this invention, diffusion filter 12c which prepared 12d of platinum coating coats in an outlet side echo and diffuser 12 is prepared. Therefore, said platinum coat 13b, the hydrogen which reached to the way among case body 12a through bore 12e in the non-contact state, and oxygen gas become that there is almost no bypassing into moisture gas outlet path 3c as it is, and it is radical-ized by contacting 12d of platinum coating coats. That is, while hydrogen and oxygen gas under the condition of radical[ non-]-izing are radical-ized by 12d of platinum coating coats of said diffusion filter 12c and hydrogen and oxygen of a non-radical-ized condition will be in the condition of \*\*\*\*\* 0, the hydrogen and oxygen which were radical-ized react in an instant, and water is generated.

[0033] moreover, the hydrogen and oxygen in the condition of the probability bypassed into moisture gas outlet path 3c while the hydrogen and oxygen in the condition of having been radical-ized when diffusion filter 12c existed in a way among said case bodies 12 have been unreacted having become smaller, and having been radical-ized -- all will contribute to a moisture generation reaction mostly.

[0034] In addition, that the platinum coating coat 13 is locally heated by heat of reaction by forming the member 8 for gaseous diffusion which consists of said entrance-side reflector 9, a filter 10, and an outlet side echo and diffuser 12 in a reactor body becomes

that there is nothing. It is proved that moisture generating can be performed where the whole region is mostly held to the temperature of the platinum coating coat 13 in which about about 500 degrees is, and it moreover continues under the high moisture generating conversion and the responsibility exceeding about 99% at insurance, and water generating of the amount of 1000 or more cc/min can be performed.

#### EXAMPLE

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[Example] In the reactor body 1 of drawing 1 , the dimension of the furnace body members 2 and 3 was made into diameter 134mmphi and the product with a thickness of 33.5mm made from SUS316L, and the curvature side of hollow parts 2a and 3a was made into the curvature side with a radius of curvature of  $R=108\text{mm}$ . Moreover, the filter (about 1.7mm in thickness) which has an average of 2.0-micrometer bore which carried out two or more sheet laminating of the mesh made from stainless steel as a filter 10 was used. That whose height the outer diameter of case object 9a is 22mmphi, and is 5mm as an entrance-side reflector 9 again furthermore, as an outlet side echo and diffuser 12 22mmphi and height 10.5mm, [ the outer diameter of case object 12a ] Diffusion filter 12c used the thing of a filter (about 1.7mm in thickness) which has an average of 200-micrometer bore which carried out two or more sheet laminating of the mesh made from a filter (about 1.7mm in thickness), and stainless steel which has an average of 70-micrometer bore which carried out two or more sheet laminating of the mesh made from stainless steel. In addition, 12d of platinum coating coats of diffusion filter 12c forms a platinum coat with a thickness of about 2 micrometers on the barrier layer anodic oxide coating made from TiN with a thickness of about 2 micrometers.

[0036] What formed barrier layer anodic oxide coating (about 2-micrometer [ in thickness ], ion plating method) 13a made from TiN in the internal surface of the furnace body member 3, and, on the other hand, formed with a thickness of about 1 micrometer platinum coat (vacuum deposition method) 13b on it as a platinum coating coat 13 was used.

[0037] Moisture generating conversion was searched for by supplying the material gas of  $\text{H}_2$  1000 cc/min+ $\text{O}_2$  1000 cc/min from gas supply path 4a,  $\text{H}_2$  1000 cc/min+ $\text{O}_2$  500 cc/min, and  $\text{H}_2$  1500 cc/min+ $\text{O}_2$  500 cc/min, and surveying the moisture which flows out of moisture gas outlet path 5a using the above-mentioned reactor for moisture generating. Consequently, even if it was in which case of the aforementioned \*\*, \*\*, and \*\*, in the continuation moisture generating trial over about 10 hours, 99.3% or more of moisture generating conversion was acquired.

#### DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is drawing of longitudinal section of the \*\* reactor for moisture generators concerning the embodiment of this invention.

[Drawing 2] It is the enlarged vertical longitudinal sectional view of an outlet side echo and diffuser.

[Drawing 3] It is I-I \*\*\*\*\* of drawing 2 .

[Drawing 4] It is drawing of longitudinal section of the reactor for moisture generating concerning point \*\*.

[Drawing 5] It is the curve which shows the moisture generating conversion of the reactor for moisture generating concerning point \*\*.

[Drawing 6] In the reactor for moisture generating of drawing 4 , it is the curve which shows the moisture generating conversion at the time of removing an outlet side reflector.

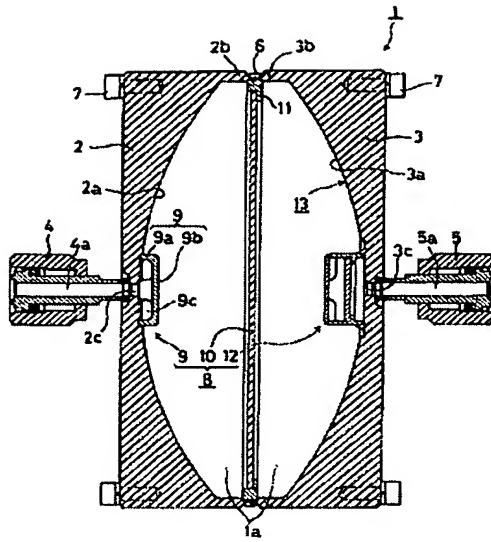
[Brief Description of Notations]

In the space section and 2, a furnace body member and 2a a hollow part and 2b A flange object, [ 1 ] [ a reactor body and 1a ] In 2c, a gas supply path and 3 a hollow part and 3b for a furnace body member and 3a A flange object, 3c the joint for gas supply, and 4a for a moisture gas outlet path and 4 A gas supply path, 5 a moisture gas outlet path and 6 for the joint for moisture gas derivation, and 5a A filter flange, In 7, the bolt for reactor mounting and 8 an entrance-side reflector and 9a for a gaseous diffusion member and 9 A case object, In 9b, a reflecting plate and 9c a filter, and 11a and 11b for a bore and 10 A filter presser foot, 12 -- an outlet side echo and diffuser, and 12a -- a case object and 12b -- a reflecting plate and 12c -- a diffusion filter and 12d -- a platinum coating coat and 12e -- a bore and 12f -- a filter presser foot and 13 -- a platinum coating coat and 13a -- barrier layer anodic oxide coating and 13b -- a platinum coat.

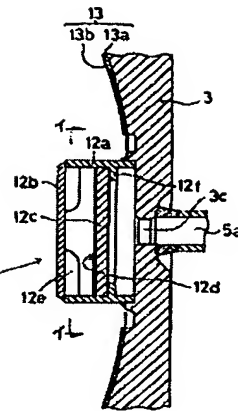
DRAWINGS

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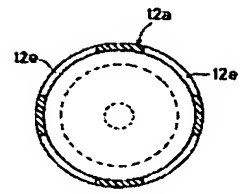
【図 1】



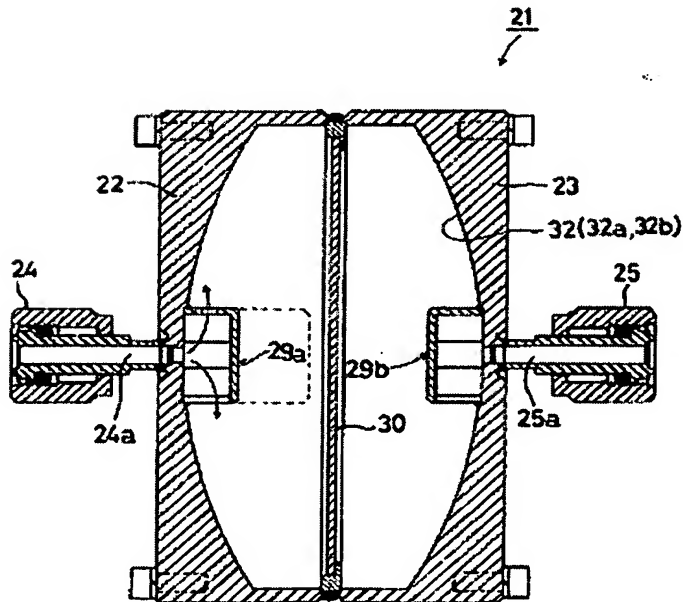
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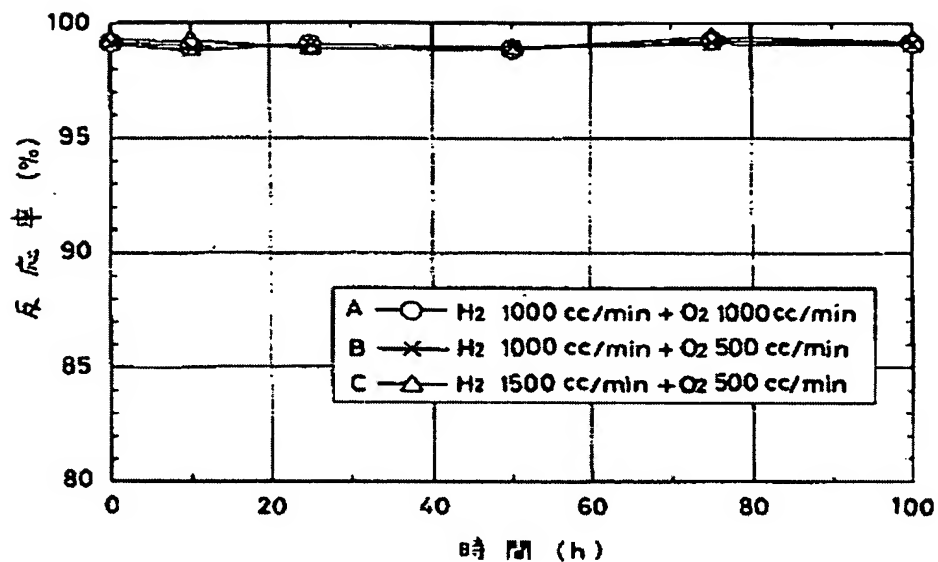
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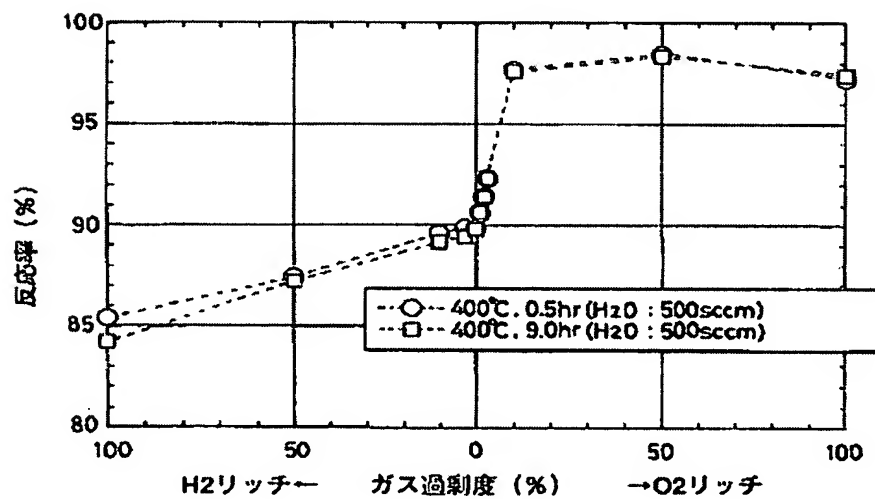
【図 4】



【図5】



【図6】





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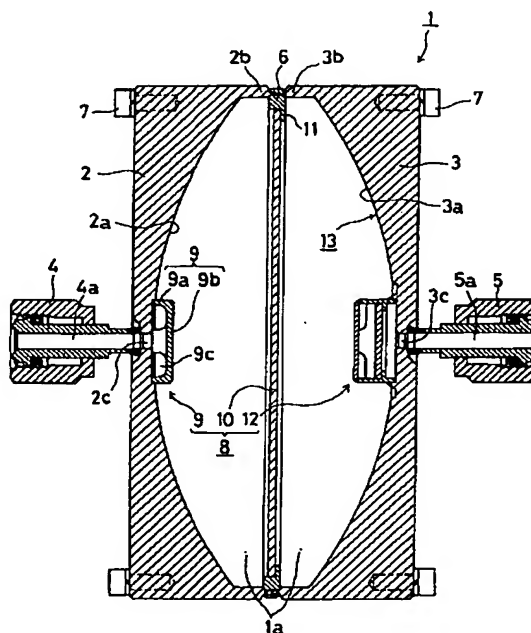
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(54) 【発明の名称】 水分発生用反応炉

(57) 【要約】

【課題】 水分発生用反応炉の小形化を図ると共に、水分発生反応率を一層高めて、未反応ガスを減少させる。

【解決手段】 二つの炉本体部材を組合せて形成され、内部に空間部を有する反応炉本体と；一方の炉本体部材に穿設され、空間部へ原料ガスを導入するガス供給通路と；他方の炉本体部材に穿設され、空間部から生成水を導出する水分ガス出口通路と；ガス供給通路と同軸状に一方の炉本体部材の空間部側に固着され、壁面に透孔を有する筒状のケース体とケース体の内側端面を閉鎖する反射板とから成る入口側反射体と；反応炉本体の空間部に配設したフィルタと；水分ガス出口通路と同軸状に他方の炉本体部材の空間部側に固着され、壁面に透孔を有する筒状のケース体とケース体の内側端面を閉鎖する反射板とケース体の内部に配設した拡散フィルタとから成る出口側反射・拡散体と；反応炉本体の内壁面に設けた白金コーティング皮膜と；から構成した水分発生用反応炉を構成する。



【特許請求の範囲】

【請求項1】 二つの炉本体部材(2)、(3)を組合せて形成され、内部に空間部(1a)を有する反応炉本体(1)と；一方の炉本体部材(2)に穿設され、前記空間部(1a)へ原料ガスを導入するガス供給通路(2c)と；他方の炉本体部材(3)に穿設され、前記空間部(1a)から生成水を導出する水分ガス出口通路(3c)と；前記ガス供給通路(2c)と同軸状に炉本体部材(2)の空間部側に固着され、壁面に透孔(9c)を有する筒状のケース体(9a)とケース体(9a)の内側端面を閉鎖する反射板(9b)とから成る入口側反射体(9)と；前記反応炉本体(1)の空間部(1a)内に配設したフィルタ(10)と；前記水分ガス出口通路(3c)と同軸状に炉本体部材(3)の空間部側に固着され、壁面に透孔(12e)を有する筒状のケース体(12a)とケース体(12a)の内側端面を閉鎖する反射板(12b)とケース体(12a)の内部に配設した拡散フィルタ(12c)と拡散フィルタ(12c)に設けた白金コーティング皮膜(12d)とから成る出口側反射・拡散体(12)と；反応炉本体(1)の内壁面に設けた白金コーティング皮膜(13)と；から構成した水分発生用反応炉。

【請求項2】 フィルタ(10)を、 $200\mu\text{m}$ 以下の透孔を有するフィルタ(10)とした請求項1に記載の水分発生用反応炉。

【請求項3】 拡散フィルタ(12c)を、 $50\mu\text{m}$ 以上の透孔を有する拡散フィルタ(12c)とした請求項1に記載の水分発生用反応炉。

【請求項4】 反応炉本体(1)を、ほぼ同形態の彎曲面状の窪部(2a)を有する炉本体部材(2)と彎曲面状の窪部(3a)を有する炉本体部材(3)とを対向状に組合せて形成すると共に、両本体部材(2)、(3)の中央部にフィルタ(10)を配設する構成とした請求項1に記載の水分発生用反応炉。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、主として半導体製造設備で用いる水分発生用反応炉の改良に関するものである。

【0002】

【従来の技術】例えば、半導体製造に於ける水分酸化法によるシリコンの酸化膜付では、少なくとも $1000\text{cc}/\text{min}$ を越える超高純度水を必要とする。そのため、本件出願人は先きに図4に示す構造の水分発生用反応炉を開発し、特願平8-242246号として公開している。

【0003】上記図4の反応炉本体21は、ガス供給用継手24及び水分ガス取出用継手25を備えた耐熱性の炉本体部材22、23と、反応炉21の内部に両炉本体部材22、23のガス供給通路24a及び水分ガス出口

通路25aと対向状に設けた入口側反射板29a及び出口側反射板29bと、反応炉21の内部中央に設けたフィルタ30と、炉本体部材23の内壁面に設けた白金コーティング皮膜32等とから形成されている。また、前記白金コーティング皮膜32は、炉本体部材23の内壁面に形成したTiN等の窒化物からなるバリヤー皮膜32aの上に、蒸着工法やイオンプレーティング工法等によって白金皮膜32bを固着することにより形成されている。

【0004】而して、ガス供給通路24aを通して反応炉本体21の内部へ供給された水素及び酸素は、入口側反射板29a、フィルタ30及び出口側反射板29bから成る拡散用部材によって拡散され、白金コーティング皮膜32と接触する。白金コーティング皮膜32と接触した酸素及び水素は、白金の触媒作用によって反応性が高められ、所謂ラジカル化された状態となる。ラジカル化された水素と酸素は、水素混合ガスの発火温度よりも低い温度下で瞬時に反応をし、高温燃焼をすることなしに水分を生成する。

【0005】前記図4の反応炉本体21は、水分発生装置の大幅な小型化が図れ、しかもより高い反応性と応答性の下で $1000\text{cc}/\text{min}$ を越える量の高純度水や高純度水と酸素との混合ガスを得ることができ、半導体製造技術の分野に於いて画期的な注目を集めているものである。

【0006】図5は、前記図4の反応炉本体(外径約 $134\text{mm}\phi$ 、厚さ $70\text{mm}$ 、内容積約 $490\text{cc}$ 、水分発生量 $1000\text{cc}/\text{min}$ 、炉温度約 $400^\circ\text{C}$ )21に於ける水分発生反応率の経時変化を示すものであり、原料ガスが酸素リッチ又は水素リッチなガスであっても、約98.5～99.0%の水分発生反応率の下で水を安定して生成することができる。

【0007】しかし、反応炉本体21の温度が約 $400^\circ\text{C}$ 以下、水分発生量が $1000\text{cc}/\text{min}$ 以上の条件下に於いては、前記水分発生反応率を約99.0%以上に上昇させることは困難であり、約1%程度の未反応の酸素や水素が生成した水分中へ混入することになる。その結果、水素や酸素を含まない純水のみ又は水素を含まない純水と酸素のみの混合物を取り出すことができないと云う問題がある。

【0008】

【発明が解決しようとする課題】本発明は、前記図4の反応炉本体21での水素と酸素の反応率をより一層高めることを課題とするものであり、反応炉本体21の温度を約 $400^\circ\text{C}$ 以下、水分発生量を $1000\text{cc}/\text{min}$ 以上の条件下に於いて、99%以上の水分発生反応率を安定且つ長期に得ることを可能にした水分発生用反応炉を提供せんとするものである。

【0009】

【課題を解決するための手段】ところで、図4の反応炉

本体21に於いて、水分ガス出口通路25aへ未反応の水素や酸素が混入してくる原因としては、①白金コーティング皮膜32と接触せず、直接に水分ガス出口通路25aへ酸素や水素が到達する場合と、②一坦はラジカル化されたものの、水素又は酸素と反応することなしに水分ガス出口通路25aへ到達し、ここでラジカル化される前の状態に戻る場合の2通りが考えられるが、前者のケースが圧倒的に多いと想定されている。

【0010】本願発明者等の実験結果によれば、図4の反応炉本体21で出口側反射板29bを取り除いた場合には、図6に示すように反応炉の温度が400℃、水分発生量が500cc/min、ガス過剰度が0の条件下に於ける水分発生反応率は、約91%となる。この反応率は、水分発生量が異なるため全く同じ条件下のデータではないが、前記図5の場合の水分発生反応率(約98%)に比較して、ほぼ7%ほど低い値となっている。

【0011】このことは、出口側反射板29bが無い場合には、相当量の酸素や水素がラジカル化されずに直接に水分ガス出口通路25aへ到達することを示しており、出口側反射板29bに改良を加えることにより、水分発生反応率の向上が可能なることを示すものである。

【0012】また、前記図6からも明らかなように、出口側反射板29bが無い場合には、原料ガスが水素リッチになるほど水分発生反応率が低下する。例えば、反応炉温度が400℃、500cc/minの水分発生量に於いて水素が100%リッチの場合には、水分発生反応率が約86%であるのに対して、酸素が100%リッチの場合には約97%となり、両者の間に約11%ほどの差が生ずる。

【0013】即ち、図4のような構造の反応炉本体21の内部に於いては、酸素の方は比較的拡散され易く、直線的走行性が小さいのに対し、水素の方は比較的拡散され難く、直線的走行性が高いため、水素リッチの原料ガスの場合には、水素の直線状の流れに酸素が随伴し、ラジカル化されずに水分ガス出口通路25aへ到達する酸素が増加するものと想定される。

【0014】そこで、本発明者は図4の反応炉本体21に於いて、出口側反射板29bのガスの拡散性、特に水素に対する拡散性を高めることにより、酸素リッチの原料ガスのみならず水素リッチの原料ガスの場合でも、水分発生反応率を図5の場合の反応率約98~99%よりも高くできることを着想した。また、この着想に基づいて各種の構造の出口側反射板・拡散板を製作すると共に、これを用いて数多くの水分発生試験を行なった。

【0015】本願発明は、上述の如き着想と、これに基づく水分発生試験の結果から創作されたものであり、請求項1に記載の発明は、炉本体部材2と炉本体部材3とを組合せて形成され、内部に空間部1aを有する反応炉本体1と；炉本体部材2に穿設され、前記空間部1aへ原料ガスを導入するガス供給通路2cと；炉本体部材3

に穿設され、前記空間部1aから生成水を導出する水分ガス出口通路3cと；前記水分ガス出口通路2cと同軸状に炉本体部材2の空間部側に固着され、壁面に透孔9cを有する筒状のケース体9aとケース体9aの内側端面を開鎖する反射板9bとから成る入口側反射板9と；前記反応炉本体1の空間部1a内に配設したフィルタ10と；前記水分ガス出口通路3cと同軸状に炉本体部材3の空間部側に固着され、壁面に透孔12eを有する筒状のケース体12aとケース体12aの内側端面を開鎖する反射板12bとケース体12aの内部に配設した拡散フィルタ12cと拡散フィルタ12cに設けた白金コーティング皮膜12dとから成る出口側反射・拡散体12と；反応炉本体1の内壁面に設けた白金コーティング皮膜13とを、発明の構成要件とするものである。

【0016】請求項2に記載の発明は、請求項1の発明に於けるフィルタ10を、200μm以下の透孔を有するフィルタ10としたものである。

【0017】請求項3に記載の発明は、請求項1の発明に於ける拡散フィルタ12cを、50μm以上の透孔を有する拡散フィルタ12cとしたものである。

【0018】請求項4に記載の発明は、請求項1の発明に於ける反応炉本体1を、ほぼ同形態の彎曲面状の窪部2aを有する炉本体部材2と彎曲面状の窪部3aを有する炉本体部材3とを対向状に組合せて形成すると共に、両本体部材2、3の中央部にフィルタ10を配設する構成としたものである。

【0019】

【発明の実施の形態】以下、図面に基づいて本発明の実施態様を説明する。図1は本発明に係る水分発生用の反応炉本体の縦断面図である。また、図2は出口側反射・拡散体の拡大断面図、図3は図2のイーイ視断面図である。図1に於いて、1は反応炉本体、2、3は炉本体部材、4はガス供給用継手、5は水分ガス取出用継手、6はフィルタフランジ、7は反応炉取付け用ボルト、8はガス拡散用部材、9は入口側反射体、10はフィルタ、11はフィルタフランジのフィルタ受け片、12は出口側反射・拡散体、13は白金コーティング皮膜であり、反応炉1は二個のほぼ同形態に形成されたステンレス鋼製炉本体部材2、3を気密状に溶接することにより、短円筒形に形成されている。

【0020】前記一方の炉本体部材2は、その内部に底面が彎曲面状の窪部2aが設けられており、更に中央部には、ガス供給通路2cが穿設されている。また、外側面にはガス供給用継手4が設けられており、この外側面に設けたガス供給用継手4のガス供給通路4aが窪部2a内へ連通されている。同様に、他方の炉本体部材3は、その内部に底面が彎曲面状の窪部3aが設けられており、更に、中央部には、ガス供給通路3cが穿設されている。また、外側面には水分ガス取出用継手5が設けられており、この外側面に設けた水分ガス取出用継手5

の水分ガス出口通路5 aが窪部3 a内へ連通されている。

【0021】前記両炉本体部材2、3の内側面には、フランジ体2 b、3 bが夫々形成されており、フィルタフランジ6を介して両フランジ体2 b、3 bを気密状に溶接固定することにより、内部に空間部1 aを有する反応炉本体1が構成されている。尚、図1では両フランジ体2 b、3 bを溶接により固着する構成としているが、両フランジ体2 b、3 bをガスケット（図示省略）を介設してクランプ（図示省略）等により解離自在に組付け固着する構成としてもよい。また、図1では両本体部材2、3をほぼ同一形状のものに形成しているが、一方を有底の筒状体の形態に、他方を筒状体の開口部を閉鎖するフランジ状の形態に形成してもよいことはもちろんである。

【0022】前記ガス拡散用部材8は入口側反射板9とフィルタ10と出口側反射・拡散板12等から形成されており、図1に示す如く反応炉本体1の内部に配設されている。即ち、入口側反射板9は短筒状のケース体9 aと、ケース体9 aの内側端面を閉鎖する反射板9 bとから形成されており、ケース体9 aの外周壁には透孔9 cが形成されている。尚、当該入口側反射板9は炉本体部材2の底面のガス供給通路2 cと対向する位置にこれと同軸状に配置され、これに溶接固着されている。

【0023】また、前記フィルタ10は、約200  $\mu$ m以下の透孔を有するステンレス鋼製フィルタより形成されており、本実施形態では平均2  $\mu$ mのメッシュ状の透孔を有するフィルタが使用されている。尚、フィルタ10の外周縁にはステンレス鋼製のフィルタフランジ6が溶接されており、このフィルタフランジ6を介してフィルタ10は炉本体部材2、3へ溶接固定されている。

【0024】更に、前記出口側反射・拡散体12は、図2及び図3に示す如く、短円筒状のケース体12 aと、ケース体12 aの内側端面を閉鎖する反射板12 bと、拡散フィルタ12 cと、拡散フィルタ12 cの空間部側の外表面に設けた白金コーティング皮膜12 dと、フィルタ押え12 f等から形成されており、ケース本体12 aの内側部分の外周壁には複数の透孔12 eが穿設されている。即ち、前記ケース体12 aや反射板12 b等は全てステンレス鋼により形成されており、反射板12 bはケース体12 aへスポット溶接により固着されている。また、拡散フィルタ12 cは50  $\mu$ m以上の透孔を有するステンレス鋼製フィルタより形成されている。

【0025】前記拡散フィルタ12 cの空間部側の外表面には、厚さ0.2～8  $\mu$ mの白金コーティング皮膜12 dが形成されている。即ち、当該白金コーティング皮膜12 dは、拡散フィルタ12 cの外表面に形成した厚さ0.1～5  $\mu$ mのTiN製のバリアー皮膜とその上に形成した厚さ0.1～3  $\mu$ mの白金皮膜とから形成されており、白金コーティング皮膜12 dの形成によって拡

散フィルタ12 cに目詰まりが生ずるのを防止するため、拡散フィルタ12 cを構成するステンレス鋼製フィルタのメッシュ状の透孔は、50  $\mu$ m以上の大きさ（本実施形態では70  $\mu$ mと200  $\mu$ m）に選定されている。

【0026】尚、白金コーティング皮膜12 dの形成方法等については、後述する反応炉本体1の内壁面に設けた白金コーティング皮膜13の場合と同様であるため、ここでは詳細な説明を省略する。また、本実施形態では、白金コーティング皮膜12 dを拡散フィルタ12 cの空間部側の外表面に形成するようにしているが、拡散フィルタ12 cの内部に白金コーティング皮膜12 dを設けることも可能である。更に、前記出口側反射・拡散体12は炉本体部材3の底面の水分ガス出口通路5 aと対向する位置に同軸状に配置され、これに溶接固着されている。

【0027】前記図1においては、炉本体部材2、3の各底面を彎曲面状としているが、これを平面状の底面に形成するようにしてもよい。また、図1に於いては、入口側反射体9や出口側反射・拡散体12の長さ寸法を窪部2 aの深さ寸法の約1/6及び窪部3 aの深さ寸法の約1/3としているが、当該長さ寸法を大きくして、フィルタ10の中心部を透過するガス量を押えるようにすることも可能である。更に、図1においては、フィルタ10としてディスク型で且つその全面をガス透過部としたフィルタを使用しているが、これに替えて、ディスク型であって且つその外周面部のみをフィルタ部（ガス透過部）とした構成のフィルタを用いるようにしてもよい。

【0028】前記白金コーティング皮膜13は、SUS316L製の炉本体部材3の内表面の全域に形成されており、先ず炉本体部材3の内表面にTiN製のバリアー皮膜13 aを形成したあと、その上に白金皮膜13 bが形成されている。また、バリアー皮膜13 aの厚さは0.1  $\mu$ m～5  $\mu$ m程度が最適であり、図1に於いては、約2  $\mu$ mの厚さのTiN製のバリアー皮膜13 aがイオンプレーティング工法により形成されている。更に、前記白金皮膜13 bの厚さは0.1  $\mu$ m～3  $\mu$ m位が適当であり、図1に於いては約1  $\mu$ mの厚さの白金皮膜13 bが真空蒸着法により形成されている。

【0029】尚、バリアー皮膜13 aの形成方法としては、前記イオンプレーティング工法以外に、イオンスパッタリング法や真空蒸着法等のPVD法や化学蒸着法（CVD法）、ホットプレス法、溶射法等を用いることも可能である。また、白金皮膜13 bの形成方法は、前記真空蒸着法以外に、イオンプレーティング工法やイオンスパッタリング法、化学蒸着法、ホットプレス法等が使用可能であり、更に、バリアー皮膜13 aがTiN等の導電性のある物質の時にはメッキ法も使用可能である。

【0030】ガス供給用継手4のガス供給通路4aを通して入口側反射体9のケース体9a内へ噴射されたガスは反射板9bへ衝突したあと、外周壁に設けた透孔9cを通して噴射され、窪部2a内で拡散されることによりフィルタ10のほぼ全面を均等に通過し、炉本体部材3の窪部3a内へ入る。また、前記窪部3a内へ噴射された水素と酸素の混合ガスは、白金コーティング皮膜13の全面に亘って均等に衝突接触し、これにより所謂触媒活性化されることになる。更に、活性化された水素と酸素は主として窪部3a内で瞬時に反応し、水を生成する。そして、主として窪部3aで形成された水分ガスは、出口側反射・拡散体12の透孔12e及び拡散フィルタ12cを通して水分ガス出口通路5aへ導出されて行く。

【0031】ところで、フィルタ10を透過して窪部3a内へ入った水素及び酸素ガスの大部分は、白金皮膜13bと衝突・接触することによりラジカル化され、ラジカル化された水素と酸素は、そのほぼ全量が瞬時に反応して水に変換される。また、窪部3a内へ入った水素及び酸素ガスの一部はそのまま直進するかも知れないが、これ等の直進した水素及び酸素ガスは反射板12bへ衝突して再拡散される。その結果、白金皮膜13bと非接触のままで透孔12eを通過して拡散フィルタ12cへ到達する水素及び酸素は、大幅に減少する。

【0032】一方、本発明に於いては、出口側反射・拡散体12内に白金コーティング皮膜12dを設けた拡散フィルタ12cを設けている。そのため、前記白金皮膜13bと非接触のままで透孔12eを通してケース本体12aの内方へ到達した水素や酸素ガスが、そのまま水分ガス出口通路3c内へ素通りすることはほぼ皆無となり、白金コーティング皮膜12dと接触することによりラジカル化される。即ち、非ラジカル化の状態下にある水素や酸素ガスは、前記拡散フィルタ12cの白金コーティング皮膜12dによってラジカル化され、非ラジカル化状態の水素や酸素が殆んど零の状態になると共に、ラジカル化された水素と酸素は瞬時に反応をし、水が生成される。

【0033】また、前記ケース本体12の内方に拡散フィルタ12cが存在することにより、ラジカル化された状態の水素と酸素が未反応のままで水分ガス出口通路3c内へ素通りする確率がより小さくなり、ラジカル化された状態の水素と酸素のほぼ全部が水分生成反応に寄与することになる。

【0034】加えて、前記入口側反射体9、フィルタ10及び出口側反射・拡散体12から成るガス拡散用部材8を反応炉本体内に設けることにより白金コーティング皮膜13が反応熱によって局部的に加熱されることが皆無となり、白金コーティング皮膜13のほぼ全域を約500°位の温度に保持した状態で水分発生を行なうことができ、約99%を越える高い水分発生反応率と応答

性の下に、安全にしかも継続して1000cc/min以上の量の水発生を行えることが実証されている。

#### 【0035】

【実施例】図1の反応炉本体1に於いて、炉本体部材2、3の外形寸法を直径134mmφ、厚さ33.5mmのSUS316L製とし、且つ窪部2a、3aの彎曲面を曲率半径R=108mmの彎曲面とした。また、フィルタ10として、ステンレス製メッシュを複数枚積層した平均2.0μmの透孔を有するフィルタ（厚さ約1.7mm）を使用した。更に、入口側反射体9として、ケース体9aの外径が22mmφ、高さが5mmのものを、また、出口側反射・拡散体12として、ケース体12aの外径が22mmφ、高さが10.5mm、拡散フィルタ12cがステンレス製メッシュを複数枚積層した平均70μmの透孔を有するフィルタ（厚さ約1.7mm）及びステンレス製メッシュを複数枚積層した平均200μmの透孔を有するフィルタ（厚さ約1.7mm）のものをを使用した。尚、拡散フィルタ12cの白金コーティング皮膜12dは、厚さ約2μmのTiN製バリアー皮膜の上に、厚さ約2μmの白金皮膜を形成したものである。

【0036】一方、白金コーティング皮膜13としては、炉本体部材3の内壁面にTiN製のバリアー皮膜（厚さ約2μm、イオンプレーティング法）13aを形成し、その上に厚さ約1μmの白金皮膜（真空蒸着法）13bを形成したものをを使用した。

【0037】上記水分発生用反応炉を用いて、ガス供給通路4aから①H<sub>2</sub> 1000cc/min+O<sub>2</sub> 1000cc/min、②H<sub>2</sub> 1000cc/min+O<sub>2</sub> 500cc/min、③H<sub>2</sub> 1500cc/min+O<sub>2</sub> 500cc/minの原料ガスを供給し、水分ガス出口通路5aから流出する水分を実測することにより、水分発生反応率を求めた。その結果、前記①、②及び③の何れのケースにあっても、約10時間に亘る連続水分発生試験に於いて、99.3%以上の水分発生反応率が得られた。

#### 【0038】

【発明の効果】本発明は上述の通り、反応炉本体の内部に入口側反射体とフィルタと出口側反射・拡散体を設けると共に、出口側反射・拡散体の内部に白金コーティング皮膜を備えた拡散フィルタを設ける構成としている。その結果、水分ガス出口通路内へ流出する未反応ガスがほとんど零となり、酸素リッチの原料ガスの場合は勿論のこと水素リッチの原料ガスの場合でも、99.0%以上の高い水分発生反応率が得られる。また、反応炉本体内の白金コーティング皮膜が反応熱によって局部的に加熱されることも皆無となり、ほぼ500℃程度の温度下で1000cc/min以上の水分を安定して発生することができる。

【0039】請求項4の発明に於いては、ほぼ同一形状

の炉本体部材を対向状に組み合わせて反応炉本体を形成する構成としている。その結果、反応炉本体の構造が簡素化され、製造コストの大幅な引下げが可能となる。本発明は上述の通り優れた実用的効用を奏するものである。

【図面の簡単な説明】

【図１】本発明の実施態様に係る水分発生装置用反応炉の縦断面図である。

【図２】出口側反射・拡散体の拡大縦断面図である。

【図３】図２のイーイ視断面図である。

【図４】先願に係る水分発生用反応炉の縦断面図である。

【図５】先願に係る水分発生用反応炉の水分発生反応率を示す曲線である。

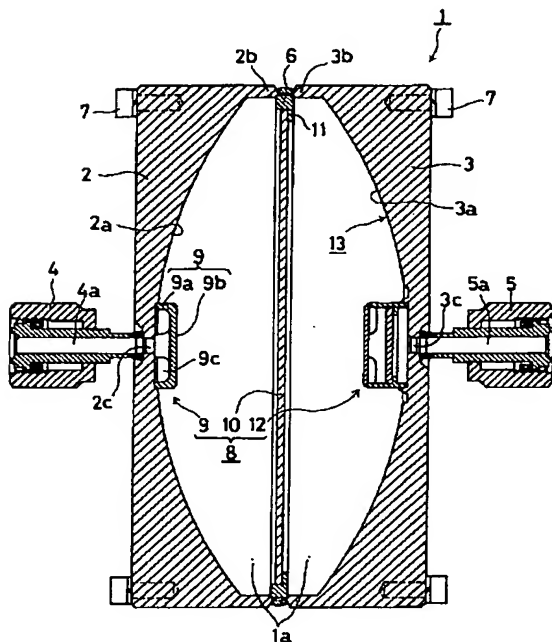
【図６】図４の水分発生用反応炉に於いて、出口側反射体を取り除いた場合の水分発生反応率を示す曲線である。

る。

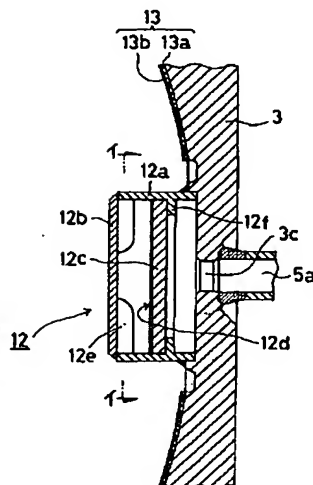
【符号の簡単な説明】

１は反応炉本体、１ａは空間部、２は炉本体部材、２ａは腔部、２ｂはフランジ体、２ｃはガス供給通路、３は炉本体部材、３ａは腔部、３ｂはフランジ体、３ｃは水分ガス出口通路、４はガス供給用継手、４ａはガス供給通路、５は水分ガス導出用継手、５ａは水分ガス出口通路、６はフィルタフランジ、７は反応炉取付用ボルト、８はガス拡散部材、９は入口側反射体、９ａはケース体、９ｂは反射板、９ｃは透孔、１０はフィルタ、１１、１１ａ・１１ｂはフィルタ押え、１２は出口側反射・拡散体、１２ａはケース体、１２ｂは反射板、１２ｃは拡散フィルタ、１２ｄは白金コーティング皮膜、１２ｅは透孔、１２ｆはフィルタ押え、１３は白金コーティング皮膜、１３ａはバリアー皮膜、１３ｂは白金皮膜。

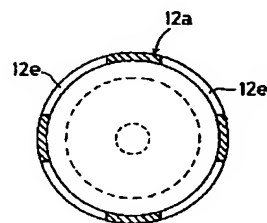
【図１】



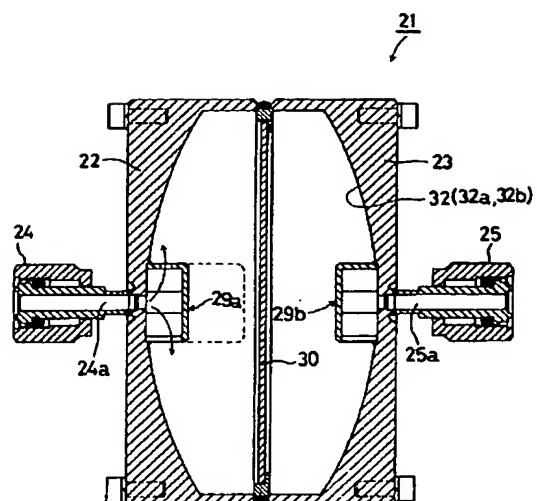
【図２】



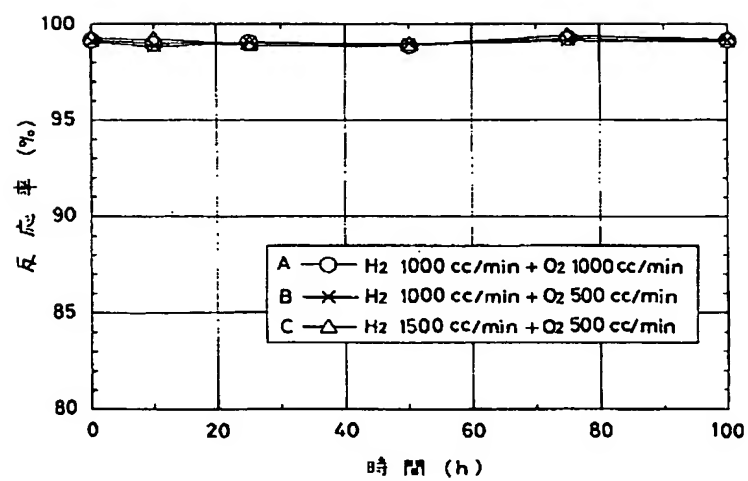
【図３】



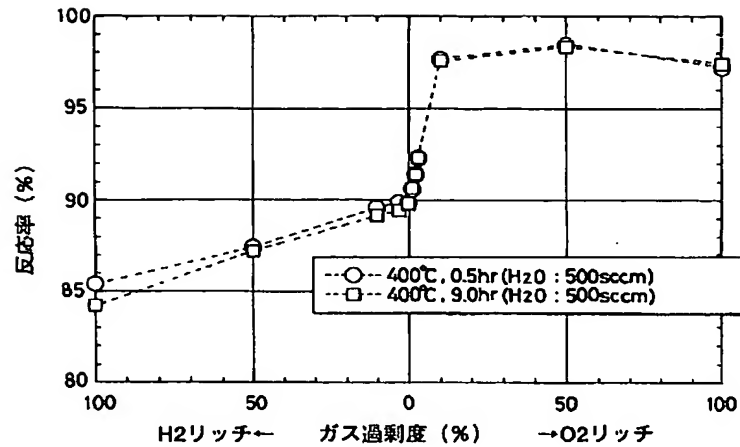
【図 4】



【図 5】



【図6】



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